
फिप्रोनिल + थियामेथोक्सम सस्पेन्शन
कन्सन्ट्रैट — विशिष्टि

Fipronil + Thiamethoxam
Suspension Concentrate —
Specification

ICS 65.100.10

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Fipronil + Thiamethoxam suspension concentrate (SC) is used as insecticide in agriculture. Fipronil + Thiamethoxam SC formulation is generally manufactured to contain a mixture of 4.0 percent (*m/m*) of fipronil and 4.0 percent (*m/m*) of thiamethoxam.

In the preparation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and rules, wherever applicable.

The composition of the Committee responsible for the formulation of this standard is given in Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FIPRONIL + THIAMETHOXAM SUSPENSION CONCENTRATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Fipronil + Thiamethoxam suspension concentrate.

2 REFERENCES

The standards listed in Annex A contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 REQUIREMENTS**3.1 Constituents**

3.1.1 The material shall consist of a suspension of fine particles of fipronil and thiamethoxam, technical, in an aqueous phase together with suitable formulants.

3.1.2 Fipronil, technical conforming to IS 18389 and thiamethoxam, technical conforming to IS 15983 shall be employed in the formulation of the material.

3.2 Physical**3.2.1 Description**

The material shall be of off white to cream homogenous liquid free from any visible impurities.

3.2.2 Pourability

When determined by the method prescribed in Annex C, the rinsed residue shall not be more than 1 percent (*m/m*).

3.2.3 Spontaneity of Dispersion

When determined by the method prescribed in Annex D, the spontaneity of dispersion of the

material shall not be less than 70 percent (*m/m*).

3.2.4 Suspensibility

When determined by the method prescribed in Annex E, the suspensibility of the material shall not be less than 75 percent (*m/m*).

3.2.5 Persistent Foam

When determined by the method prescribed in Annex F, the persistent foam shall not be more than 60 ml in 12 min.

3.2.6 Wet Sieve Test

Not less than 98 percent by mass of the material shall pass through a 45 micron IS sieve when determined by method described in Annex G.

3.3 Chemical

The material shall comply with the chemical requirements specified in **3.3.1** and **3.3.2**.

3.3.1 When determined by the method prescribed in Annex B, the observed fipronil and thiamethoxam content, percent (*m/m*) of any of the samples shall not differ from the declared nominal value by more than tolerance limits indicated below:

Nominal Value, percent	Tolerance, percent	
Up to 9	+10 - 5	} of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 - 3	

The actual value of fipronil and thiamethoxam content in the formulation shall be calculated to the second decimal place and then rounded off to first decimal place before applying the tolerance given in **3.3.1**

3.3.2 Acidity/Alkalinity

When tested by the method prescribed in IS 6940, the acidity (as H_2SO_4) or alkalinity (as NaOH) of the material shall not be more than 0.5 percent by mass, respectively.

4 PACKING

The material shall be packed in high density polyethylene (HDPE) containers/bottles of capacities 100 ml, 250 ml, 500 ml and 1 litre. The HDPE containers for packing of material shall conform to IS 9754. These shall be further packed in 5 ply corrugated boxes of 10×1 litre, 20×500 ml, 40×250 ml. The corrugated fibreboard boxes shall conform to IS 2771 (Part 1). The containers shall also conform to the various general requirements stipulated in IS 8190 (Part 2).

5 MARKING

5.1 The containers shall bear legibly and indelibly the following:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Batch number;
- d) Date of manufacture;
- e) Date of expiry;
- f) Net quantity;
- g) Nominal fipronil content, percent (m/m);
- h) Nominal thiamethoxam content, percent (m/m);
- j) A cautionary notice as worded in the

Insecticide Act, 1968, and rules framed there under; and

- k) Any other information required under the *Legal Metrology (Packaged Commodities) Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10627.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred in 3.2.2 to 3.2.6, 3.3.1 and 3.3.2.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)		pesticides (<i>second revision</i>)
IS 2771 (Part 1) : 2022	Corrugated fibreboard boxes — Specification: Part 1 General requirements (<i>third revision</i>)	IS 9754 : 1981	High density polyethylene containers for packing of liquid pesticides (upto 1 litre capacity)
IS 6940 : 1982	Methods of test for pesticides and their formulations (<i>first revision</i>)	IS 10627 : 1983	Methods for sampling of pesticidal formulations
		IS 15983 : 2013	Thiamethoxam, technical — Specification
IS 8190 (Part 2) : 1988	Requirement for packing of pesticides: Part 2 Liquid	IS 18389 : 2023	Fipronil, technical — Specification

ANNEX B

(Clause 3.3.1)

DETERMINATION OF ACTIVE INGREDIENTS IN FIPRONIL + THIAMETHOXAM SC

B-1 GENERAL

Two methods (Method I and Method II) have been specified for determination of active ingredients in Fipronil + Thiamethoxam SC. Both the methods use high performance liquid chromatography technique. In Method I, the active ingredients in the formulation are determined in separate run while in Method II, both the ingredients in the formulation are determined in a single run. Method II shall be referee method in case of any dispute.

B-2 METHOD I

B-2.1 Determination of Fipronil Content in Fipronil + Thiamethoxam SC

B-2.1.1 Apparatus

B-2.1.1.1 High performance liquid chromatography equipped with a constant flow pump, sample injector capable of injecting 20 µl aliquots and an ultraviolet detector coupled to chromatography software and printer is used for this determination:

Instrument	HPLC with UV detector and suitable chromatography software
Column	Silica, 25 cm × 4.6 mm i.d., 5 µm particle size
Mobile Phase	2 percent isopropyl alcohol in cyclohexane
Flow Rate	1.2 ml/min.
Injection Volume	20 µl
Detector	Ultraviolet detector (280 nm)

B-2.1.1.2 Analytical balance

B-2.1.1.3 Microlitre syringe — 25 µl capacity

B-2.1.1.4 Ultrasonic bath

B-2.1.1.5 Standard glassware

B-2.1.2 Reagents

B-2.1.2.1 Isopropyl alcohol — HPLC grade

B-2.1.2.2 Cyclohexane — HPLC grade

B-2.1.2.3 Fipronil reference standard — of known purity

B-2.1.3 Procedure

B-2.1.3.1 Preparation of the standard solution

Weigh about 0.1 g of fipronil standard in 50 ml of volumetric flask. Add isopropyl alcohol and dilute upto the mark. Then pipette out 10 ml of above solution, transfer it in a 50 ml of volumetric flask and dilute with cyclohexane upto the mark.

B-2.1.3.2 Preparation of sample solution

Weigh about a quantity of sample equivalent to 0.1 g of fipronil in 50 ml of volumetric flask. Add isopropyl alcohol and dilute upto the mark. Then pipette out 10 ml of above solution, transfer it in a 50 ml of volumetric flask and dilute with cyclohexane upto the mark.

B-2.1.3.3 Inject the standard solution (**B-2.1.3.1**) and the sample solution (**B-2.1.3.2**) reference and test sample solutions in replicate till consistent readings are obtained and calculate the fipronil content as given in **B-2.1.4**.

B-2.1.4 Calculation

Fipronil content, percent by mass =

$$\frac{A_2 \times M_1}{M_2 \times A_1} \times P$$

where

- A_2 = peak area of fipronil in sample solution;
 M_1 = mass, in g, of standard fipronil;
 P = percentage purity of standard;
 M_2 = mass, in g, of sample taken for test; and
 A_1 = peak area of fipronil in standard solution.

B-2.2 Determination of Thiamethoxam Content in Fipronil + Thiamethoxam SC

B-2.2.1 Apparatus

B-2.2.1.1 High performance liquid chromatograph equipped with a constant flow pump, sample injector capable of injecting 20 µl aliquots and an ultraviolet detector coupled to chromatography

software and printer is used for this determination.

Instrument	HPLC with UV detector and suitable chromatography software
Column	ODS C 18, (25 cm × 3.9 mm i.d., 5 µ).
Mobile Phase	Acetonitrile : Water : Phosphoric acid (85 percent) (30 : 70 : 0.35 v/v)
Flow Rate	1.0 ml/min
Injection Volume	20 µl
Detector	Ultraviolet detector (280 nm)

B-2.2.1.2 Analytical balance

B-2.2.1.3 *Microtitre syringe* — 25 µl capacity

B-2.2.1.4 *Ultrasonic bath*

B-2.2.1.5 *Standard glassware*

B-2.2.2 Reagents

B-2.2.2.1 *Phosphoric acid* — AR grade

B-2.2.2.2 *Acetonitrile* — HPLC grade

B-2.2.2.3 *Water* — HPLC grade

B-2.2.2.4 *Thiamethoxam reference standard* — of known purity

B-2.2.3 Procedure

B-2.2.3.1 Preparation of the standard solution

Weigh about 0.08 g of thiamethoxam reference standard and dilute to 50 ml with acetonitrile. Dilute 5 ml of this solution to 100 ml with mobile phase.

B-2.2.3.2 Preparation of sample solution

Weigh an amount of sample equivalent to 0.08 g of thiamethoxam and dilute to 50 ml with acetonitrile. Dilute 5 ml of this solution to 100 ml with mobile phase.

B-2.2.3.3 Inject the standard solution (**B-2.2.3.1**) and the sample solution (**B-2.2.3.2**) in replicate till consistent readings are obtained and calculate the thiamethoxam content as given in **B-2.2.4**.

B-2.2.4 Calculation

Thiamethoxam content, percent by mass =

$$\frac{M_1 \times A_2}{M_2 \times A_1} \times P$$

where

M_1	=	mass, in g, of thiamethoxam standard;
A_2	=	area of thiamethoxam peak in the sample;
P	=	percentage purity of standard;
M_2	=	mass of thiamethoxam sample; and
A_1	=	area of thiamethoxam peak in the standard.

B-3 METHOD II

B-3.1 Determination of Fipronil and Thiamethoxam Contents in Fipronil + Thiamethoxam SC

B-3.1.1 Apparatus

B-3.1.1.1 High performance liquid chromatograph equipped with a constant flow pump, sample injector capable of injecting 20 µl aliquots and an ultraviolet detector coupled to chromatography software and printer is used for this determination.

Instrument	HPLC with UV detector and suitable chromatography software
Column	Nucleosil C18 (5 µm); 250 mm × 4.6 mm i.d., or equivalent.
Mobile Phase	Acetonitrile + Water (70 + 30)
Flow Rate	1.0 ml/min
Injection Volume	20 µl
Detector	Ultraviolet detector (280 nm)
Temperature	Ambient
Retention time	Fipronil — 8.0 min approx. Thiamethoxam — 3.0 min approx.

B-3.1.1.2 Analytical balance

B-3.1.1.3 *Microtitre syringe* — 25 µl capacity

B-3.1.1.4 Ultrasonic bath

B-3.1.1.5 Standard glassware

B-3.1.2 Reagents

B-3.1.2.1 Standard reference — of known purity

B-3.1.2.2 Acetonitrile — HPLC grade

B-3.1.2.3 Water — HPLC grade

B-3.1.3 Procedure

B-3.1.3.1 Preparation of standard solution

Weigh about 0.05 g of fipronil standard and 0.05 g of thiamethoxam standard into volumetric flask (50 ml) and add 25 ml acetonitrile into flask and place the flask in an ultrasonic bath for approximately 5 min. Allow it to attain room temperature and make up the volume up to the mark with acetonitrile and mix thoroughly.

Pipette out 5.0 ml of solution into a 25 ml volumetric flask and makeup to the mark with acetonitrile and homogenize.

B-3.1.3.2 Preparation of sample solution

Weigh a quantity of sample so as to contain 0.05 g of fipronil and 0.05 g of thiamethoxam standard into 50 ml volumetric flask and add 25 ml of acetonitrile

into flask and place the flask in an ultrasonic bath for approximately 5 min. Allow it to attain room temperature and make up the volume up to the mark with acetonitrile and mix thoroughly.

Pipette out 5.0 ml of solution into a 25 ml volumetric flask and makeup to the mark with Acetonitrile and homogenize.

B-3.1.3.3 Inject the standard solution (**B-3.1.3.1**) and the sample solution (**B-3.1.3.2**) in replicate till consistent readings are obtained and calculate the active ingredients as given in **B-3.1.4**.

A-3.1.4 Calculation

Fipronil/thiamethoxam content, percent by mass =

$$\frac{M_1 \times A_2}{M_2 \times A_1} \times P$$

where

M_1	=	mass of fipronil/thiamethoxam standard;
A_2	=	area of fipronil/thiamethoxam peak in the sample;
A_1	=	area of fipronil/thiamethoxam peak in the standard;
M_2	=	mass of fipronil/thiamethoxam sample; and
P	=	percentage purity of fipronil/thiamethoxam standard.

ANNEX C

(Clause 3.2.2)

DETERMINATION OF POURABILITY IN FIPRONIL + THIAMETHOXAM SC

C-1 OUTLINE OF THE METHOD

The suspension concentrate is allowed to stand for a definite time and the amount remaining in the container after a standardized pouring procedure is determined. The container is rinsed and the amount then remaining is determined.

C-1.1 Apparatus/Equipment

C-1.1.1 A 500 ml stoppered measuring cylinder as per following requirements:

- a) Volume equivalent to 1 : 5 ml subdivision of the scale
- b) Capacity corresponding : 50 ml to lowest graduation mark
- c) Capacity corresponding : 500 ml to highest graduation mark
- d) Length of scale : 250 mm
- e) Overall height : 39 cm
- f) Diameter of base : 10 cm
- g) Stopper : B 34

NOTE — High density polyethylene bottles, 1 000 ml volume and Kilner jars, 700 ml volume, can be used but this must be recorded with the result.

C-1.2 Procedure

Weigh the empty container and stopper (W_0) and add enough of the test item taken from a recently mixed bulk sample to leave approximately 20 percent of the volume of the container as ullage. Replace the stopper and reweigh the container (W_1).

Allow the container to stand undisturbed for 24 h and then pour out the test item (*see* Note 1) for 60 s at an angle of 45° and then finally invert the container for 60 s (*see* Note 2). Re-weigh the container and stopper (W_2). Add distilled water at 20°C (a volume of 80 percent of that of the container) and replace the stopper. Invert the container 10 times (*see* Note 3) and empty the container as before and re-weigh the container and stopper (W_3). Calculate the residue (R) and the rinsed residue (R_1).

C-1.3 Calculation

$$\text{Residue (R), percent by mass} = \frac{W_2 - W_0}{W_1 - W_0} \times 100$$

Rinsed residue (R_1), percent by mass =

$$\frac{W_3 - W_0}{W_1 - W_0} \times 100$$

where

W_2 = mass, in g, of the container after the pouring out of the suspension concentrate;

W_0 = mass, in g, of the empty container;

W_1 = mass, in g, of the container with the sample; and

W_3 = mass, in g, of the container after removal of distilled water.

NOTES

1 The length of the standing period and the temperature should be agreed previously.

2 A square sided container should be held so that a flat side is underneath.

3 The term invert the container means that the container's vertical axis is turned through 180 °C and then brought back to its original position, the whole operation taking about 2 s.

ANNEX D

(Clause 3.2.3)

DETERMINATION OF SPONTANEITY OF DISPERSION IN FIPRONIL + THIAMETHOXAM SC**D-1 OUTLINE OF THE METHOD**

The method is broadly similar to that used to determine the suspensibility of concentrates, except that it employs only one inversion and a 5 min standing time. It involves preparing 250 ml of a mixture of formulation and water, mixed only with one inversion of the cylinder. After standing under defined conditions the top nine-tenth is removed, and the remaining tenth assayed chemically or by solvent extraction. The spontaneity of dispersion is readily calculated.

D-2 REAGENT

Standard Hard Water

D-3 APPARATUS

Graduated cylinders, glass stoppered, 250 ml capacity. Glass suction tubes about 40 cm long, 5 mm internal diameter (i.d.) drawn out at one end to 2 mm to 3 mm internal diameter (i.d.).

D-4 PROCEDURE

Normally the whole procedure is carried out in duplicate. Homogenize the sample by gently stirring. The standard hard water, measuring cylinder and sample to be used in the determination should be equilibrated to the required temperature before starting the test. Determine the density of the formulation and calculate the mass of formulation equivalent to 12.5 ml (W). Pour standard water (237.5 ml) at the required temperature into the graduated cylinder and stand this on a top-pan balance, tare the weight add the calculated mass of formulation from a small beaker held so that the lip is 1 cm above the top of the cylinder. Complete the

addition within a time limit of 15 s. Record the weight of the formulation added. As soon as the formulation has been added, stopper the cylinder and invert once. Stand the cylinder in an upright position on a bench free from vibration or direct sources of heat for 5 min + 10 s. At the end of this time, carefully remove the stopper and withdraw the top 225 ml of suspension by means of the suction tube connected to a reservoir and suitable pump. Carry out the operation in 10 s to 15 s by maintaining the tip of the tube just below the falling level of the suspension, care being taken to minimize any disturbance of the suspension. Ensure that the tip of the tube is always only a few mm below the surface of the suspension.

Transfer the 25 ml of dilute suspension remaining in the cylinder. Extract the active ingredient just like in suspensibility and calculate the spontaneity of dispersion. Determine the active ingredient content in the 25 ml bottom suspension.

D-5 CALCULATION

Spontaneity of dispersion, percent by mass =

$$\frac{111 (C - Q)}{C}$$

where

$C = \frac{W \times a}{100}$ = mass, in g, in the whole cylinder;

Q = mass, in g, of active ingredient in 25 ml sample at the bottom;

W = mass, in g, of formulation actually added to the cylinder; and

a = percentage of active ingredient by mass in formulation.

ANNEX E

(Clause 3.2.4)

DETERMINATION OF SUSPENSIBILITY IN FIPRONIL + THIAMETHOXAM SC

E-1 REAGENTS

Standard hard water

E-2 APPARATUS

Beaker & Glass rod

E-3 PROCEDURE (FOR FIPRONIL)

Carry out the whole procedure in duplicate. Homogenize the sample by gently stirring (see Note 1). The mass of sample to be taken is that required to make 250 ml of diluted suspension, with the concentration recommended on the label. If a range of concentrations is recommended, the highest and lowest should be used (see Note 2). The standard hard water, measuring cylinder and sample to be used in the determination should be equilibrated to the required temperature.

Place 100 ml of equilibrated standard water in a 250 ml measuring cylinder. Weigh the required amount of sample into a 50 ml beaker and transfer quantitatively to the cylinder with more standard water, finally making up to 250 ml. Stopper the cylinder and invert 30 times (see Note 3). Place the cylinder in an upright position, free from vibration, and allow it to stand undisturbed for 30 min.

At the end of the time, quickly insert the glass tube into the cylinder and by means of a vacuum pump and suitable reservoir withdraw nine-tenths (225 ml) of the suspension. Carry out the operation in 10 s to 15 s by maintaining the tip of the glass tube just below the falling level of the suspension, care being taken to minimize any disturbance of the suspension (see Note 4).

Transfer the 25 ± 1 ml of dilute suspension remaining in the cylinder to an evaporation dish and evaporate the water to dryness over a water bath. Record the weight of the residue. Find out the quantity (Q) of active ingredient in the residue by multiplying weight of residue with the assayed percent active ingredient content in the formulation (see Annex B) and dividing by 100. Calculate the suspensibility as given in E-4.

NOTES

1 A crust of dry material sometimes forms inside the neck of suspension concentrate containers. Care must be taken to ensure that particles do not break away from this and contaminate the sample used for the determination.

2 It may be helpful to weigh the beaker plus formulation before and after the addition to the water to make a check on the amount of sample added to the water.

3 To invert the cylinder, hold it with one hand at each end, insulated with a cloth, and rotate it through 180 °C and back again, through an imaginary fixed point midway between the hands. Ensure no bouncing occurs. Each inversion should take 2 s.

4 It may be helpful to fit a rubber bung, larger than the cylinder mouth, as an adjustable stop so that the final level of suspension left in the cylinder is repeatable. The position of the bung can be determined by a preliminary calibration.

E-4 CALCULATION (FOR FIPRONIL)

Suspensibility, percent by mass =

$$\frac{111 (C - Q)}{C}$$

where

$C = \frac{a \times W}{100}$ = mass, in g, in the whole cylinder;

Q = mass, in, g of active ingredient in 25 ml sample at the bottom;

a = percent by mass of active ingredient in the formulation; and

W = mass, in g, of formulation actually added to the cylinder.

E-5 PROCEDURE (FOR THIAMETHOXAM)

Carry out the whole procedure in duplicate. Homogenize the sample by gently stirring (see Note 1). The mass of sample to be taken is that required to make 250 ml of diluted suspension, with the concentration recommended on the label. If a range of concentrations is recommended, the highest and lowest should be used (see Note 2). The standard hard water, measuring cylinder and sample to be used in the determination should be equilibrated to the required temperature.

Place 100 ml of equilibrated standard water in a 250 ml measuring cylinder. Weigh the required amount of sample into a 50 ml beaker and transfer quantitatively to the cylinder with more standard water, finally making up to 250 ml. Stopper the cylinder and invert 30 times (see Note 3). Place the cylinder in an upright position, free from vibration, and allow it to stand undisturbed for 30 min.

At the end of the time, quickly insert the glass tube into the cylinder and by means of a vacuum pump and suitable reservoir withdraw nine-tenths (225 ml)

of the suspension. Carry out the operation in 10 s to 15 s by maintaining the tip of the glass tube just below the falling level of the suspension, care being taken to minimize any disturbance of the suspension (see Note 4).

Transfer the (25 ± 1) ml of dilute suspension remaining in the cylinder to an evaporation dish and evaporate the water to dryness over a water bath. Record the weight of the residue. Find out the quantity (Q) of active ingredient in the residue by multiplying weight of residue with the assayed percent active ingredient content in the formulation (see Annex B) and dividing by 100. Calculate the suspensibility as given in **E-6**.

NOTES

1 A crust of dry material sometimes forms inside the neck of suspension concentrate containers. Care must be taken to ensure that particles do not break away from this and contaminate the sample used for the determination.

2 It may be helpful to weigh the beaker plus formulation before and after the addition to the water to make a check on the amount of sample added to the water.

3 To invert the cylinder, hold it with one hand at each end,

insulated with a cloth, and rotate it through 180°C and back again, through an imaginary fixed point midway between the hands. Ensure no bouncing occurs. Each inversion should take 2 s.

4 It may be helpful to fit a rubber bung, larger than the cylinder mouth, as an adjustable stop so that the final level of suspension left in the cylinder is repeatable. The position of the bung can be determined by a preliminary calibration.

E-6 CALCULATION (FOR THAIAMET-HOXAM)

Suspensibility, percent by mass =

$$\frac{111 (C - Q)}{C}$$

where

$$c = \frac{a \times W}{100} = \text{mass, in g, in the whole cylinder;}$$

Q = mass, in, g of active ingredient in 25 ml sample at the bottom;

a = percent by mass of active ingredient in the formulation; and

W = mass, in g, of formulation actually added to the cylinder.

ANNEX F*(Clause 3.2.5)***DETERMINATION OF PERSISTANT FOAM IN FIPRONIL + THIAMETHOXAM SC****F-1 OUTLINE OF THE METHOD**

The suspension concentrate is diluted in a measuring cylinder of standard dimensions which is inverted 30 times and, the amount of foam created and remaining after certain time is measured.

F-2 APPARATUS**F-2.1 Graduated Cylinder**

Glass stoppered of 250 ml capacity, with 2 ml graduations. The distance between the 0 mark and the 250 ml mark should be 20 cm to 21.5 cm and between the 250 ml mark and the bottom of the stoppered, 6 cm to 8 cm (The cylinder should be clean and free from grease).

F-2.2 Stopwatch**F-3 REAGENT**

Standard water

F-4 PROCEDURE

The mass of sample to be taken is that mass required

to make 200 ml of a diluted formulation with a concentration recommended in the directions for use supplied with the product. When several concentrations are recommended, the maximum concentration shall be used.

Put about 180 ml of standard hard water into the 250 ml measuring cylinder standing on a top pan balance and weigh in the required amount of the sample. Fill to 200 ml with standard hard water and record the temperature (see Note 1). Stopper the cylinder and then invert it 30 times by hand through 180 degrees and back to its original position, the whole operation being completed in approximately 2 s. Place the stoppered cylinder upright on the bench and immediately start the stopwatch. Read the volume of foam produced and remaining after 12 min (see Note 2).

NOTES

1 The recommended temperature is $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

2 A few bubbles round the periphery are not significant. Any volumes above the 250 ml mark should be marked on the outside and the volume of foam thus determined.

ANNEX G

(Clause 3.2.6)

DETERMINATION OF WET SIEVE TEST IN FIPRONIL + THIAMETHOXAM SC

G-1 PRINCIPLE

The method is suitable for the determination of the amount of the non-dispersible material in Fipronil + Thiamethoxam SC.

G-2 APPARATUS

G-2.1 Test Sieve — specified IS Sieve [see IS 460 (Part 1)] prepared for the test by removing any film, grease or any other water repellent material and then by drying.

G-2.2 Rubber Hose — of 10 mm internal diameter

G-3 PROCEDURE**G-3.1 Wet Sieving**

Transfer 10 g of the sample to the sieve, rinsing with tap water. Wash the material on the sieve with an

oscillating jet of tap water. Using a rubber hose of 10 mm i.d. delivering 4 litres to 5 litres of water per min. Continue the washing for 10 min, directing the water from the circumference of the sieve towards the center and keeping the end of the hose at a distance of between 2cm to 5 cm from the surface of sieve. Transfer the residue to a pre weighed watch glass with a jet of distilled water from a wash bottle. Dry to constant weight and record the weight of the sample to the nearest 0.01 g.

G-4 CALCULATION

Material passing through the IS sieve, percent by mass =

$$100 \left(1 - \frac{m}{M}\right)$$

where

m = mass, in g, of the dry residue obtained;
and

M = mass, in g, of the material taken for test.

ANNEX H*(Foreword)***COMMITTEE COMPOSITION**

Pesticides Sectional Committee, FAD 01

<i>Organization</i>	<i>Representative(s)</i>
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR J. P. SINGH (<i>Chairperson</i>)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (<i>Alternate</i>)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (<i>Alternate</i>)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (<i>Alternate</i>)
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